Materials Science Division Project Safety Review
Safety Analysis Form (03/08)

Date of Submission	March 12, 2010	FWP No.:	58405

Project Title	User Experimental Work with Electron Microscopes in the Electron Microscopy Center This Safety Analysis Form (SAF) supersedes previous versions of 20006 and its modifications.

Is this a (check one) new submission [ ] renewal [ ] supplemental modification [X]

Principal Investigator(s) Dean Miller

Other Participants (excluding administrative support personnel) EMC staff and EMC users

(Attach participant signature sheet)

Project dates:	Start:	March 2010	End:	Open-ended

This form is to be completed for all new investigations or experimental projects that are conducted in MSD laboratories, and for all ongoing such projects that undergo significant change from their original scope of work, or where there has been an addition of a potentially new hazard not covered in the original review. It is not intended to be used for office work, routine maintenance activities, or administrative tasks.

Experimental work may not be performed until the project safety review has been completed, procedures have been approved, and the work has been authorized (ESH Manual 21.2.3).

The completed form and all supporting documentation is to be submitted to the MSD ESH Coordinator by the principal investigator with sufficient advance notice and information to allow a project safety review prior to the beginning of the experiment. The information will be reviewed by the Division Director, members of the MSD safety review team, and by outside experts (if appropriate) for unresolved safety, health, and environmental issues associated with the proposed work. The principal investigator may be asked to resolve outstanding issues through consultations with the safety review team before the work begins. The information submitted will be reviewed by an independent review team, and final approval will be granted by the Division Director.

This form must be accompanied by a participant signature form once work has been authorized.

The principal investigator must be familiar with the responsibilities of a lead experimenter and the general requirements of the experiment safety review in the Argonne ESH Manual, section 21.2.

Useful references:
Argonne ESH Manual: http://www.aim.anl.gov/manuals/eshman/
Argonne Waste Handling Procedures Manual: http://www.aim.anl.gov/manuals/whpm/
MSD Chemical Hygiene Plan: http://www.msd.anl.gov/resources/esh/
Material Safety Data Sheets: https://webapps.inside.anl.gov/cms/msds/

Table of Contents[1]:

List of Attachments:

SOPs for electron microscopes SOPs for specimen holders MSDSs for acutely toxic or carcinogenic materials Signs for SF_6 systems

[1] To update table of contents, right/command click inside table; from resulting contextual menu select "Update field", then "Update page numbers only"

1. Scope of Project (ISM Core Function 1)

1.1 General Description

Provide a general overview description of the project. While scientific background is important, concentrate on an operational description that focuses on the experimental work done in the laboratory.

This project provides and manages a suite of electron microscopes (TEM/STEMs and SEMs) in Argonne's Electron Microscopy Center (EMC), which is a Department of Energy National User Facility and whose purpose is to facilitate the pursuit of materials research by scientists and students from Argonne National Laboratory and other institutions.

The scope of this project involves attaching a specimen to a holder, inserting the holder into an electron microscope, operating the electron microscope while the specimen is inside, removing the holder when the observations or measurements are complete, and removing the specimen from the holder.

The electron microscopes in this project are used for imaging and microanalysis of materials using direct excitation of a material by electrons which have been accelerated to potentials of between 200 V to 300 kV in vacuum systems with pressures from 10^{-10} to 20 torr. Imaging and microanalysis are accomplished by measuring signals generated by the electron-specimen interaction and include absorbed, secondary, backscattered, and transmitted electrons, photons, and x-rays.

Samples/specimens are attached to various kinds of holders. Sample holders for scanning electron microscopes (SEMs) are typically of aluminum or graphite; one is a disk of Be. SEM samples are either clamped in the holders with screws or held in place with carbon tape, carbon paste, or silver paste. Heating holders (T≤1500˚C) and a Peltier-cooled holder are available for the SEM in D127.

Sample holders for transmission electron microscopes (TEMs) are made of various non-magnetic materials such as steel, copper, brass, aluminum, tantalum, and beryllium. TEM samples are held in place by clamps, threaded rings, silver or carbon paint, or glues. Specialty sample holders that are available to users for *in situ* experimentation include heating holders (T≤1000˚C), holders that can be cooled with liquid nitrogen or liquid helium, a holder that heats and strains samples, a holder that heats and applies a voltage to samples, a gas-reaction cell holder, and a holder that produces in-plane magnetic fields at the sample. Other TEM holders are owned by users and have electro-mechanical components for *in situ* measurements of friction or electrical transport.

All prospective users must submit a research proposal to obtain permission to conduct research using facility instruments. The proposal details the proposed research, the desired instruments and operating modes, any ancillary devices required, and information about the specimens to be studied. The EMC staff reviews the proposals for safety issues and technical feasibility to determine whether the proposed work is within the scope of this project's SAF. The proposals are then reviewed and scored by a Proposal Review Committee (a peer-review panel). Users must submit annual research progress reports to the EMC and submit a proposal for continuing research.

1.2 Modules of Project

Describe the various components that make up this project. Components can be pieces of equipment or specific hazardous or complex tasks within the project that require special training to use or perform safely. Indicate locations, even if the project consists of only one component. Indicate custodians for major equipment. Attach designs, drawings, or other useful descriptive material.

Components	Locations	Custodians
FEI Titan 80-300 (ACAT)	bldg. 216: rooms A107 and A108	Bernd Kabius
FEI Tecnai F20ST TEM/STEM	bldg. 212: room DL139	Russell Cook
FEI/Philips CM30T TEM	bldg. 212: room DL133	Russell Cook
FEI CM200 TEM	bldg. 212: room DL132	Nestor Zaluzec
FEI Quanta 400F ESEM	bldg. 212: room D127	Russell Cook
Hitachi H-9000 NAR TEM (IVEM)	bldg. 212: room G149	Marquis Kirk
Hitachi S-4700-II SEM	bldg. 212: room DL120	Russell Cook
Zeiss 1540XB FIB-SEM	bldg. 212: room DL135	Jon Hiller
Zeiss NVision FIB-SEM	bldg 212: room DL136	Dean Miller
Dark room for developing film	bldg. 212: room D130	Jon Hiller
Computing facility	bldg. 212: room DL238	Russell Cook
Olympus and Zeiss light microscopes	bldg. 212: room DL128	Jon Hiller
Liquid helium transfer	bldg. 212: rooms DL139 & G149	
Operations support areas	bldg. 212: rooms DL133A, DL134, DL137, DC110A, DM110, G145, G149A bldg. 216: room B105 and corridor A113.	

Each of the first nine "components" are commercial electron microscopes, and they have other commercial sub-components (detectors, stages, etc) that are manufactured by companies other than the microscope manufacturers. None of the instruments have been custom-designed or built at Argonne.

The computing facility is used for off-line analysis of images, spectra, etc.

Liquid helium is occasionally transferred from commercial 100 L dewars to TEM holders in 212/DL139 and 212/G149; the transfer process is detailed in SOPs.

The operations support areas are located near the electron microscope rooms and contain equipment that sustain the operation of the microscopes and the laboratory environment.

1.3 Project Limits

Define the range of samples, chemicals, physical conditions that you consider covered under this project review. For chemicals and samples include either specific cases that are considered extremely hazardous, e.g. silane, HF, etc., or general classes such as reactive metals, oxidizers, etc. In some cases it may be useful to define the envelope by specifically excluding certain hazard categories.

This SAF covers the operation of the electron microscopes in their standard imaging and microanalysis operating modes as described in their respective SOPs and in the instrument manufacturers' operating manuals. It includes the operation of specialized equipment and specimen holders that are owned by the EMC, all of which have EMC-approved SOPs. It also includes the use of a dark room for developing film. Light microscopes are also available to examine materials at magnifications up to 1000x.

It covers the use of user-owned specimen holders or other equipment *only after* such equipment has passed an electrical inspection, the equipment has been examined by EMC staff for instrument incompatibilities, and an SOP or SAF has been provided to the EMC for review and approval.

The typical specimens studied in these instruments consist of stable inorganic materials (metals, oxides, composites, semiconductors, superconductors, etc.) and organic materials such as polymers or bio/nano complexes. Sample sizes vary according to material and microscope. Many TEM specimens (typically 3 mm in diameter) consist of nanoparticles placed onto 20 nm-thick carbon films that are supported by 50 µm-thick metal grids. Other TEM specimens are self-supporting in that they have thick rims (~100 µm) and dimpled centers. There are many other varieties of TEM samples, but all are limited in size by the industry-standard specimen holders and the penetrating power of electrons. SEM specimens cover a larger size range, from TEM-size samples to 150 mm diameter X 27 mm thick for the Hitachi S-4700-II SEM. SEM samples are limited in size only by the stage and airlock mechanisms.

All research proposals must contain details of all specimens and declare any hazards that may be associated with the materials or with user-supplied equipment. The EMC staff reviews the proposals for hazards and, if necessary, notifies the proposers of any hazard controls (engineering, procedural, or PPE) they must put in place.

The following work or materials are excluded from this project:

1. Work or materials that are classified as biohazard level 2 (BSL2) or higher.
2. Combustible, flammable, reactive, corrosive, or unstable samples.
3. Radioactive materials are not generally allowed. The proposer must submit a separate SAF for that work because the instrument rooms are not Controlled Areas. In those rare cases in which radioactive specimens are allowed via the proposal review system, health physics technicians post the room as a Controlled Area, limit access to the room, and conduct pre- and post-experiment surveys. MSD SAF 20008 addresses the handling of weakly radioactive TEM samples in the Hitachi H-9000 NAR TEM.
4. Work that involves modifications of electron columns or detectors and that would be accomplished by someone other than authorized representatives of the instrument manufacturers.
5. Sample preparation in 212/DL126. Such work is covered under MSD SAF 20007.
6. Operation of the particle accelerators in G-wing when they are used to inject an ion beam into the specimen region of the Hitachi H-9000 NAR TEM. Accelerator operations are covered under MSD SAF 20004.
7. Operation or maintenance of equipment in the operations support areas. Only trained EMC staff, manufacturers' engineers, and FMS or contracted service personnel may operate or maintain equipment in those areas. However, users may operate a vacuum system for a specimen holder in DL133A.

2. Hazard Analysis (ISM Core Function 2)

2.1 Hazard List

Hazard/Issue	Yes	No	Unknown
Does the proposed work, as you perceive it, intrinsically contain the following safety, health, or environmental issues or concerns?			
Chemical Hazards			
Use of toxic chemicals	X		
Use of flammable chemicals	X		
Use of carcinogenic chemicals	X		
Generation of hazardous or toxic wastes	X		

Use of explosive or highly reactive chemicals		X	
Use of strong acids or bases	X		
Use of carbon monoxide gas		X	
Use of hydrogen gas (above 4% concentration)		X	
Use of perchloric acid or perchlorate salts		X	
Use of hydrofluoric acid		X	
Nanomaterials			
Nanoparticles dispersible in air	X		
Nanoparticles dispersible in liquids	X		
Biological Hazards			
Work with Biosafety Level 2 or above[2]		X	
Radiological Hazards			
Use of radioisotopes (see section 6)		X	
Exposure to ionizing radiation (excluding radioisotopes)		X	
Generation of radioactive wastes		X	
Physical Hazards			
Use of Class III or Class IV lasers		X	
Use of cryogenic fluids	X		
Use of high magnetic fields	X		
Use of high voltage or high amperage equipment	X		
Electrical work on energized equipment (>50V)		X	
Operation of equipment at high vacuums	X		
Operation of equipment at elevated pressures	X		
Use of compressed gases	X		
Operation of equipment at high temperatures	X		
Hazardous Working Environments			
Working in areas with high noise levels		X	
Potential exposure to climatic extremes		X	
Working at elevated heights		X	
Entering confined spaces		X	
Use of self-contained breathing apparatus or respirators		X	
Work in areas of mechanical hazards		X	
Other (explain)[3]			
Sharps	X		

2.2 Hazard Details

For all the hazards checked Yes or Unknown, provide specific details, including locations (unless obvious from 1.3). For highly hazardous or energetic chemicals, indicate specific chemicals, quantities used. For physical hazards, give quantitative details (e.g., voltages for electrical hazards, power and wavelengths of lasers).

Hazard	Detail
Toxic chemicals	1. Be dust is toxic. Manufacturer-supplied specimen holder cups, retaining nuts, washers, and a platen of Be are used in building 212 (DL120, DL132, DL133, DL139 and G149) and in building 216 (A107 and A108).

[2] Requires review by Institutional Biosafety Committee

[3] See ESH Manual 21.3 Appendix A for a more comprehensive list of potential hazards

	2. Phenanthrene is used in a sealed cartridge (volume: a few ml) in the FIB-SEMs in 212 (DL135 & DL136) to create carbon films on sample surfaces. It is not accessible by users. 3. Silver in the fixer recycling cartridge (not accessible by users) in the darkroom 212/D130 and in silver paint that is used with some SEM specimens in 212 (DL120, D127, DL135 and DL136). 4. Some TEM or SEM specimens may contain toxic materials (including Arsenic, Lead, Chromium (VI), Cadmium, and their compounds).
Flammable chemicals	1. 500 ml plastic squeeze bottles containing methanol, ethanol, isopropanol, or acetone may be in temporary use in any of the rooms listed in section 1.2 (except 212/DL238). 2. Pump oil in vacuum roughing pumps in 212 (DL120, D127, D130, DL132, DL133, DL137, DL139 and G149) and 216 (A107 and A113). Pump oils are in the least-hazardous class of combustible liquids (class IIIB). 3. Pentamethylcyclopentasiloxane (CAS 6166-88-5; flash point 39˚C) is used in a sealed cartridge (volume: a few ml) in the FIB-SEM in 212/DL135 to deposit SiO_2. It is not accessible by users. 4. 1,3,5,7-Tetramethylcyclotetrasiloxane (CAS 2370-88-9; flash point 25˚C) is used in a sealed cartridge (volume: a few ml) in the FIB-SEM in 212/DL136 to deposit SiO_2. It is not accessible by users.
Carcinogenic chemicals	1. Be dust is a Class 1 carcinogen. Manufacturer-supplied specimen holder cups, retaining nuts, washers, and a platen of Be are used in 212 (DL120, DL132, DL133, DL139 and G149) and 216/A107. 2. Some TEM or SEM specimens may contain carcinogens (including Arsenic, Lead, Chromium (VI), Cadmium, and their compounds). 3. Falcon/Edwal Hypo-Chek is used in the dark room 212/D130 by EMC staff to test the fixer for exhaustion. It is classified as a Class 1 carcinogen because of 1 component: formaldehyde.
Hazardous wastes	Acetone and ethanol wastes generated in microscope laboratories are collected in the Satellite Waste Accumulation Areas #21200006 (212/DL126), #21200024 (212/G235) and #21600001 (216/B105).
Acids and bases	1. Acetic acid in the Falcon/Edwal Quick-Fix fixer in 212/D130. 2. Sulfuric acid in the Falcon/Edwal Hardener in 212/D130. 3. Sodium hydroxide in the Kodak Anti-Fog in 212/D130. 4. Gallium is a corrosive and is in sealed ion sources in the FIB-SEMs in 212 (DL135 & DL136). It is not accessible by users. 5. Xenon difluoride is a corrosive and is in a sealed cartridge (volume: a few ml) in the FIB-SEMs in 212 (DL135 & DL136). It is used to etch silicon. It is not accessible by users.
Nanomaterials	A significant percentage of specimens are engineered nanomaterials. According to the Argonne Policy and Procedure Dictionary, an engineered nanomaterial (ENM) is, "An intentionally created (in contrast with natural or incidentally formed) material with one or more dimensions greater than 1 nanometer and less than 100 nanometers. An ENM is also an unbound engineered nanoparticle (UNP)."
Cryogenic fluids	1. Liquid nitrogen (LN2) is used in all of the TEM and SEM rooms for cryogenic cooling of X-ray detectors, anticontaminator devices, and two TEM specimen holders. LN2 storage dewars (100-150 L) are in 212 (D15, G147, and G149) and 216/B105. LN2 transfer dewars (4 L) are used in all of the TEM and SEM rooms.

	2. Liquid helium is used for cryogenic cooling of one TEM specimen holder in 212/DL139 and another in 212/G149. A liquid helium dewar (100 L) is stored occasionally in 212 (D15 and G147).
High magnetic fields	Within 6 inches of some ion pumps there may be magnetic fields (≥ 5 G) that might interfere with pacemakers.
High voltages	The electron guns of the existing instruments operate over the range of 200 V to 300 kV. Some electron and x-ray detectors also employ high voltage (1-2 kV) collection components.
High vacuums	Pressures from 10^{-10} to 20 torr are used in the columns of the electron microscopes that are listed in section 1.2 .
Elevated pressures	1. Air at 90 psi is supplied by building 212 and 216 compressors to the pneumatic valves on the electron microscopes and to the specimen holder storage system in 212/G145. 2. SF_6 gas is supplied to the high voltage components of TEMs at pressures ≤6 bar (90 psi). The supply cylinders are stored in 212/DL133A and 216/B105. In 212, the gas is routed through D15 to DL131, DL134 and DL137, and then into microscopes in DL132, DL133 and DL139. In 216, the gas is routed to four manifolds in A113 and then into microscopes in A101, A103, and A107. 3. Nitrogen gas at 9 psi from the building 212 nitrogen distribution system is supplied to the manifolds in 212 (DL131, DL133A, DL134 and DL137). This pressure is reduced to 1.5 psi before it is delivered to the electron microscopes and dry boxes in the microscope rooms. In 216, nitrogen gas (<15 psi) from a cylinder in B105 is routed to manifolds in A113, and the pressure is reduced to to approximately 1.5 psi before delivery to A101 , A103, and A107. 4. Liquid nitrogen storage dewars (100-150 L) are in 212 (D15, G147 and G149) and 216/B105. Pressures are approximately 20 psi. 5. Nitrogen gas (60 psi) from a cylinder is used to damp vibrations in the IVEM ion beam column in G149. 6. Nitrogen, helium, or argon may be supplied at 1.5 psi from cylinders to the specimen chamber of the SEM in 212/D127.
Compressed gases	1. Cylinders of helium gas (used with the liquid helium-cooled TEM specimen holders at 1-3 psi) are stored in DM110 and G149A. 2. SF_6 gas is supplied to the high voltage components of some TEMs at pressures ≤6 bar. The supply cylinders are stored in 212/DL133A and 216/B105. 3. A cylinder supplying nitrogen gas at 60 psi (used to damp vibrations in the IVEM ion beam column) is stored in G149A. 4. The following gases may be stored in 212/D127 to provide alternative gases for the SEM specimen chamber in that room: nitrogen, helium, argon.
High temperatures	1. Specimen heating holders are available for the FEI and Hitachi TEMs (T≤1000˚C) and for the SEM in 212/D127 (T≤1500˚C). 2. A hot air gun is available for use with the cooled specimen holders. 3. Other hot equipment: vacuum pumps, film dryer, water chiller components, some vacuum gauges.
Sharps	Tweezers, scalpels, hypodermic needles, and razor blades are used routinely in all of the TEM and SEM rooms

2.3 Waste Produced

Describe types and expected quantities of wastes produced by this project (also see 3.6 for the handling of these wastes). Address all applicable major classes (nonhazardous, hazardous/chemical, radioactive, mixed) and the specific types within these classes. Also include wastes that derive from the future decommissioning of equipment (e.g., transformer oils, accumulated debris in reaction chambers) and termination of projects (leftover chemical inventory, samples, materials).

Hazardous/flammable wastes: It is estimated that 2 liter/year of waste acetone and 2 liter/year of waste ethanol may be generated by users for cleaning small parts in conjunction with their experiments.

Samples: The TEM and SEM users must remove their specimens and materials from the EMC. Most of those samples and materials are non-hazardous except that they have been classified as nano materials (< 100 nm in at least 1 dimension).

2.4 Effluents and Emissions

Describe types and expected quantities of materials disposed into the water drains (effluent). Include process water if the amount disposed is unusually large (e.g., constantly running water). See ESH Manual 10.4 for rules regarding disposal of chemicals into the waste water stream. Consult with the building manager for stricter rules due to the condition of drain pipes.

The dark room process water – water used to wash negatives after developing and fixing (water running constantly for a total of 40 minutes) – will contain some small, unknown quantities of developer and fixer. Because the dark room is being used less and less, these amounts are decreasing.

Describe types and expected quantities of gases, aerosols, and other volatile materials emitted to the atmosphere through the ventilation system (incl. hoods)

Roughing pump exhaust gases from all microscopes in 212/D-wing are piped into the fume hood exhaust system through a HEPA filter in 212/DL126 and not into the laboratory spaces. This is a continuous process while instruments are under vacuum. Minute amounts of some chemicals from the FIB gas injection system and their reaction products are pumped to the atmosphere through the same system. Some minute amounts of SF_6 are pumped to the atmosphere through the same system when the CM30T TEM is cycled through shutdown and restart.

Are all components of this project considered "bench scale research" (NEPA)? Yes X No ____
[Limited in any single experiment, measurement, or test to 5 gal. or 5 lbs of hazardous material, or 1 lb of extremely hazardous material (40 CFR 355)]

2.5 Interaction With Other Projects

Describe possible interactions with other projects (or separate components within the same project) that are carried out in the same or adjacent spaces. Are there incompatibilities of hazards that need to be mitigated by spatial separation or staggered times of operation (e.g. lasers vs. other work in laser access controlled area). Does this project introduce major hazards into a building that are not covered under the current Building Emergency Plan (Bldg. 223 emergency plan on MSD intranet, http://www.msd.anl.gov/resources/esh/ , confer with building managers for other buildings)?

The components of this project are compatible with each other. Neither does this project introduce hazards into building 212 or 216 that are not covered under the current Building 212 or 216 Emergency Plans.

Two projects are carried out in DL126, which is adjacent to microscope room DL120. DL126 is not part of this SAF, but users of DL120 must pass through DL126. Both of the DL126 projects are compatible with this project because the users of DL120 pass by only low-hazard processes to enter DL120. The MSD-SAF-20007 project (TEM/SEM Specimen Preparation Work) project deals with sample/specimen preparation activities in 212/DL126. The MSD-SAF-20003 project is about electropolishing TEM samples using a chilled 5% perchloric acid in methanol solution in the northeast hood, which is roped-off and away from the path to DL120.

The MSD-SAF-200004 project (MSD Accelerator Lab) in the 212/G-wing facility is compatible with this project.

The MSD-SAF-200008 project (Handling Weakly Radioactive TEM Samples at the IVEM-Tandem User Facility) in the 212/G-wing facility is compatible with this project.

The EMC requires users to submit separate SAFs or SOPs when it becomes clear during the proposal review that their materials, work parameters, or equipment are either outside the scope of this SAF or a detailed SOP is necessary. Those SAFs or SOPs are then scrutinized for incompatibilities with this SAF and solutions are put in place before work can commence.

Maintenance activities in any of the microscope rooms or adjacent spaces may be compatible with experimental work. When such activities will affect experimental work on one or more microscopes, those instruments are reserved for service work so that users will not attempt to use them. The spaces affected by service or maintenance are posted and cordoned-off as necessary.

3. Hazard Control (ISM Core Function 3)

3.1 Design Features and Engineering Controls

For all hazards present, describe the design features and engineering controls applied to control the hazards. Engineering controls include enclosures and barriers that cannot be removed without the use of tools, interlocks, ventilation, software controls, etc. Engineering controls are possible and should be first line of control for all hazard classes (chemical, physical, electrical, biological, radiological).

Toxic chemicals:

1. Manufacturer-supplied specimen holder cups, retaining nuts, washers, & platens of Be are used in 212 (DL120, DL132, DL133, DL139 and G149) and 216 (A107 and A108). ES&H Manual section 4.6.5.2 exempts such finished articles from the Chronic Beryllium Disease Program because they do not release Be dust or otherwise result in exposure to airborne concentrations of Be under normal conditions of use.
2. Phenanthrene is in a sealed cartridge (volume: a few ml) and it is not accessible by users.
3. Silver in the fixer recycling cartridge is not accessible by users.
4. Although some TEM and SEM specimens contain toxic materials, they are designed so that they do not create dusts that can be inhaled. This includes Arsenic, Lead, Chromium (VI), Cadmium, and their compounds.

Flammable chemicals:

1. Hoods are available in 212/DL126 and 212/G147 for cleaning items that require large amounts of acetone or alcohols. *Note:* use of the 212/DL126 laboratory is governed by MSD SAF 20007.

2. Pentamethylcyclopentasiloxane and 1,3,5,7-tetramethylcyclotetrasiloxane are used in sealed cartridges (volume: a few ml) in the FIB-SEMs in 212 (DL135 & DL136). They are not accessible to users.
3. Vacuum pump oil is contained within the pumps and is inaccessible by users.

Carcinogenic chemicals:
1. See comment above regarding Be finished articles.
2. Although some TEM and SEM specimen materials contain carcinogens, they are designed so that they do not create dusts that can be inhaled. This includes Arsenic, Lead, Chromium (VI), Cadmium, and their compounds.

Acids and bases:
1. Gallium (a corrosive) is in sealed ion sources and it is not accessible by users.
2. Xenon difluoride (a corrosive) is in sealed cartridges (volume: a few ml) and is not accessible by users. The FIB-SEM in 212/DL136 houses the cartridge in a cabinet with $CaCO_3$ sorbent.

Nanomaterials: These must be prepared *in the users' laboratories* for TEM or SEM examination, including placing them on TEM grids or SEM stubs (with tape or paint or paste). *It is forbidden to blow compressed gas on such samples in the EMC.* If it is necessary to prepare these materials in 212/G147, the permission of the IVEM Facility Coordinator must be obtained in advance so that work may be prepared and conducted in accordance with LMS-PROC-83. The use of 212/DL126 for such work is governed by MSD-SAF-20007 (TEM/SEM Specimen Preparation Laboratory). Once the materials are mounted on SEM stubs with tape or paint or paste or onto TEM grids, they are no longer dispersible.

Cryogenic fluids: Storage dewars are equipped with relief valves, rupture discs, and vent valves.

High magnetic fields: High magnetic fields of the electron microscopes are contained within the instruments. Only within 6 inches of some ion pumps may there be magnetic fields ≥ 5 G (measured by EMC staff) that might interfere with pacemakers. There are cages surrounding the ion pumps to keep people away from them, and microscope users do not approach the ion pumps that closely if they follow the SOPs.

High voltages and high vacuums: Electron microscopes are commercial high voltage, high vacuum instruments, with ionizing radiation produced internally. The manufacturers have built engineering controls into their instruments using safety switches, shielding, and other safeguards to protect users from high voltages, X-ray radiation, high vacuum failures, etc. Users have no access to any electrical connections at high voltages. Some electron and x-ray detectors also employ high voltage (1-2 kV) collection components. These are similarly inaccessible to users when energized.

Elevated pressures and compressed gases:
1. Pressure systems are equipped with relief and vent valves.
2. SF_6 is used in the high voltage tanks and electron gun systems of the FEI TEMs in 212 (DL132, DL133, DL139) and 216/A107. SF_6 can decompose to fluorine gas (highly corrosive) and sulfur oxides in the presence of an ignition source or temperatures higher than 250˚C, such as during a fire. Such an event should be mitigated by the smoke detection and fire suppression systems. The amounts of SF_6 in each microscope room have been calculated to be insufficient to be asphyxiation hazards.

High temperatures: With the exception of some vacuum pumps, a hot air gun, and the interior

of the film dryer, hot surfaces are not generally accessible (specimen heating holders, water chiller components, ion gauges, etc.).

3.2 Procedural Controls

For complex hazardous tasks, describe how the hazards are controlled by the work procedure. E.g., specific order of tasks, verification of instrument readings, required use of special tools, and the like.

Acids and bases: The fixer working solution in the darkroom 212/D130 is mildly acidic (dilute acetic and sulfuric acids). The posted procedure for developing films tells users to don nitrile gloves so that their hands will not come into contact with the solution. *Liquid helium transfers:* The SOP for transferring liquid helium to the cooled specimen holders provides a specific order in which the tasks must be performed. The SOP includes a list of the PPE to be worn, the requirement that two people must be present, and the requirement that the laboratory doors must be open during transfers to prevent an asphyxiation hazard. People are trained and supervised in this procedure by EMC staff members. *High temperatures:* The SOPs for the TEM and SEM specimen heating holders provide a specific order in which the tasks must be performed. The TEM SOP specifically states that the holder is not to be removed from the TEM until it has been cooled to a specific temperature. The SOP for the SEM specimen heating holder specifically states that the vacuum chamber is not to be vented until the holder has been cooled to a specific temperature. People are trained and supervised in these procedures by EMC staff members.

3.3 Personal Protective Equipment

List personal protective equipment (PPE) to be worn. Be specific to task or situation, unless it applies to all laboratory work in this project. Remember that safety glasses are not sufficient splash protection against certain chemicals. For gloves, be specific as to type appropriate for the task.

Task/Situation	Personal Protective Equipment
All laboratories	Safety glasses (ANSI Z87.1 compliant). *Exceptions: safety glasses are not required when the user is simply operating an electron microscope, looking through the eyepiece lenses of light microscopes, or evaluating data with the computers and no other activities are occurring.*
Transferring liquid helium and liquid nitrogen	Cryogenic gloves (supplied by EMC), full face shield (supplied by EMC), full-length trousers without cuffs, closed-toe shoes with non-absorbent-material uppers (i.e. leather), lab coat with buttons fastened (or long-sleeve shirt with cuffs buttoned and the shirt tail outside the trousers).
Working with acetone and alcohols to clean small parts	Nitrile gloves (supplied by EMC).
Developing film in 212/D130	Nitrile gloves (supplied by EMC).
Handling TEM or SEM specimens containing nano, toxic, or carcinogenic materials	Nitrile gloves (supplied by EMC.

3.4 Training

Indicate the training required for participation in this project. Include Argonne-supplied training (list course numbers and titles), job-specific training (indicate who provides training, how records are kept), and external training (academic requirement, specialized training and/or certification).

External training: 1. People who wish to operate TEMs must have at least one college-level course in TEM with a lab component or previous TEM experience. The college course can't be one in which TEM

was just one of many topics.
2. Prospective SEM users must have similar qualifications or they must read a document which the EMC will provide and achieve a grade of 80% on the written test of that material.

Argonne-supplied training:
The APS Users Office initiates a Job Hazards Questionnaire (JHQ) and a Training Profile for those users who are not Argonne employees but who will be working in the EMC more than 10 days per year. The JHQ and training profile are generated automatically when a user registers with the Argonne User Facilities On-Line Registration System at http://www.anl.gov/Science_and_Technology/userreg.html . Users are then able to take the required courses on-line before they arrive at the EMC. The required Argonne courses for this project are in the following list and are available to registered users at http://www.aps.anl.gov/Safety_and_Training/Training/users_nonresident.html .

- EMC101, EMC Users Orientation
- ESH100U, ANL User Facility Orientation (or ESH100)
- ESH223U, Cyber Security Program Training (or ESH223)
- ESH377, Electrical Safety Awareness

Job-specific training:
Prospective microscope users must receive on-the-job training for each microscope that they will use. That training is provided by an EMC staff member using the instrument-specific SOP and other materials as seems necessary. Safety has been incorporated into the SOPs by restricting the users to certain operations, by written/posted warnings, and by written instructions for off-normal or emergency situations. Trainees must go through a rigorous period of familiarization under the supervision of a designated user, and they must demonstrate to the responsible EMC staff member that they can operate each instrument according to its SOP before they will be qualified as a user. The EMC staff member is responsible for determining when the prospective user is qualified to operate the microscope.

The EMC user administration maintains database records of users' on-the-job training.

3.5 Chemical Storage

Describe the specific locations where chemicals and gases are stored including type of storage (e.g., flammables cabinet) and how hazardous chemicals are labeled. Include precautions taken for the storage of carcinogens. Indicate who is responsible for keeping the Chemical Management System (barcodes) up-to-date. Attach Material Safety Data Sheets (MSDS) for the particularly hazardous chemicals, and describe where all MSDS are available near the location of the project. The PI must ensure that MSDS for all chemicals used in this project are on file in the Chemical Management System (https://webapps.inside.anl.gov/cms/msds/) and that all participants have ready access to them.

Chemicals:
1. Flammable liquids are stored in the flammable hazards cabinet in 212/DL126 and in the cabinet under the fume hood in 212/G147 (<5 gallons total). Vacuum pump oil may be stored in individual microscope laboratories (<5 gallons total).
2. The chemicals for film development are stored in the 212/D130 dark room.

Gases:
1. SF_6 gas cylinders are stored in 212/DL133A and 216/B105.
2. Helium gas cylinders are stored in 212 (D127, DM110, and G149A).
3. Nitrogen gas cylinders are stored in 212 (D127, G149A, DL133A) and 216/B105.
4. Argon gas cylinders are stored in 212 (D127 and DM110).

Carcinogens: Falcon/Edwal Hypo-Chek is classified as a Class 1 carcinogen because of 1 component: formaldehyde. It is stored in 212/D130 in a cabinet in secondary containment.

Hazardous chemicals are labeled with NFPA diamond information.

Jon Hiller is responsible for keeping the CMS barcodes up-to-date in 212/D-wing, and Peter Baldo is responsible for keeping the CMS barcodes up-to-date in 212/G-wing.

Hard copies of MSDSs are kept in binders on top of the flammable hazards cabinet in 212/DL126, in a binder in 212/G143, and in a binder in the desk in 216/B107.

3.6 Sample Storage and Disposition

Describe how and where samples utilized and produced in this project are stored (and labeled!) while not in active use, how long they will be retained, and how they will be disposed of.

In general, users' samples are stored in the participants' own laboratory space when not being studied in one of the EMC microscopes. The participants are responsible for removing their specimens and materials from the EMC.

Specimen storage is provided for EMC staff in dry boxes in the microscope rooms or in cabinets or drawers in 212/G151. Samples are stored in labeled specialty boxes, small glass bottles, and plastic boxes of various sorts. Labels must include the researcher's name, the chemical composition (not a shorthand identification) of the material, and the date.

Samples are retained as long as the researchers need them. When the proposals of these researchers expire, they must remove their materials from tthe EMC laboratories. When EMC staff members terminate their employment, they must remove their materials from the EMC laboratories. These requirements are enforced by the EMC's safety representative and the EMC director.

If the samples are not hazardous, the researchers may dispose of them in the trash. Otherwise, they must dispose of their samples in accordance with federal and state regulations. It is their responsibility to determine the proper disposal method.

3.7 Waste Handling

For all hazardous, radioactive, and mixed wastes, describe where and how they are accumulated (include satellite waste area number), and who will be responsible for writing up the waste for disposal by Waste Management. Indicate any special circumstances (special containers, venting, etc.) regarding the safe storage of waste. Address the prevention of incompatible waste mixtures. Include plans for dealing with the waste produced by the future decommissioning of equipment and termination of projects.

In 212/D-wing, hazardous waste is kept in the Satellite Waste Accumulation Area # 21200006 in 212/DL126. Jon Hiller is the custodian and either he or Russell Cook submit the waste requisition forms.

In 212/G-wing, hazardous waste is kept in the Satellite Waste Accumulation Area # 21200024 in 212/G233. Peter Baldo is the custodian and he submits the waste requisition forms.

In 216, hazardous waste is kept in the Satellite Waste Accumulation Area # 21600001 in 216/B105. Russell Cook is the custodian and he submits the waste requisition forms.

Incompatible wastes are not mixed: collection containers are labeled with the type of waste that may be put into it.

3.8 Emergency Management

If this project involves chemical hazards, esp. the use of corrosive chemicals, list locations of eyewash stations and safety showers. Indicate who is responsible for checking eyewash stations weekly.

Three eyewash stations are in 212 (D130, DL126 and G147). Jon Hiller is responsible for checking the eyewash stations in 212/D-wing and Peter Baldo is responsible for checking the eyewash station in 212/G147. One eyewash station is in 216/B105. FMS personnel are responsible for checking its operation. Two safety showers are in corridor 212/D15.

Include emergency procedures in case of accidents, evacuations, or other hazardous situations. Include egress routes into common areas (hallways), safe shutdown procedures, and other pertinent information. Procedures may be attached. Are all hazard categories posted at the laboratory doors?

The EMC101 EMC Users Orientation Course contains emergency procedures and maps showing exits from the facilities. SOPs document the emergency procedures for each instrument. Hazard category lists are posted on laboratory doors.

3.9 Additional Hazard Control

Describe here any measures of hazard controls that are not already documented in the previous sections.

None

3.10 Guidance Documents

List all documents, publications, and books, that you have consulted in the hazard analysis and control. Include relevant chapters and sections of the ES&H Manual but do not include those chapters that are requirements documents for other documents (e.g., 4.2). The divisional Chemical Hygiene Plan (http://www.msd.anl.gov/resources/esh/) is mandatory reading for all participants in projects that contain chemical hazards.

Hazard	Guidance
Toxic chemicals	Argonne ES&H Manual sections 4.1 & 4.3; MSD Chemical Hygiene Plan
Flammable chemicals	Argonne ES&H Manual sections 11.3 & 4.3
Carcinogenic chemicals	Argonne ES&H Manual section 4.5
Hazardous wastes	relevant MSDS; Argonne Waste Handling Procedures Manual; MSD Chemical Hygiene Plan
Strong acids or bases	Argonne ES&H Manual section 4.3; MSD Chemical Hygiene Plan; relevant MSDS
Nanomaterials	Argonne LMS-PROC-83
Cryogenic fluids	Argonne ES&H Manual section 4.10
High magnetic fields	Argonne ES&H Manual section 6.3.5
High voltage equip.	Argonne ES&H Manual sections 9.1 & 9.3
High vacuum equip.	Argonne ES&H Manual section 13.1
Elevated pressures	Argonne ES&H Manual sections 13.1 and 4.3.11
Compressed gases	Argonne ES&H Manual sections 13.1 & 13.2
High temperatures	None.
Sharps	ESH Manual section 4.3.5

4. Working Within Controls (ISM Core Function 4)

4.1 List of Work Procedures

List all work procedures relevant to this project

Standard operating procedures for each electron microscope
SOPs for specialized accessories, specimen holder, detectors, etc.
Manufacturers' operating instructions
Posted instructions concerning PPE, Be sample holders, etc.
Posted lists of approved users with work restrictions

4.2 Dosimetry

List locations where radiation dosimeters must be worn. Indicate if a ring is required in addition to the regular badge, and whether neutron dosimetry (type BGN) or not (type BG) is required. Consult with Health Physics regarding requirements.

Location	Dosimetry Requirement
None	None. Health Physics technicians survey the electron microscopes yearly. The measured X-ray radiation emanating from the instruments is below background when the shielding is properly maintained.

Identify individuals who will be issued dosimeters.

Name	Ring (Y/N)	Neutrons (Y/N)
None		

4.3 Safety Monitoring Equipment

Describe any equipment that is used to monitor safe working conditions (e.g., oxygen monitors, background radiation alarms). Note that all such equipment must be approved by Industrial Hygiene (or Health Physics for radiological monitoring).

Health Physics places area dosimeters on the microscopes and on the west wall of DL120. The EMC owns a Ludlum 177-35 G-M counter for surveying instruments for stray X-ray radiation. This instrument is calibrated annually by Health Physics.

4.4 Industrial Hygiene Monitoring

List the periodic Industrial Hygiene sampling that is required based on chemical, biological, or other hazardous materials used in this project.

None

4.5 Medical Surveillance

Identify individuals who will be placed in a medical surveillance program as a result of their participation in this project.

Argonne employees using Arsenic, Lead, Chromium (VI), Cadmium, and their compounds will be identified via JHQ questions C6.3, C6.8, C6.10 ((chromate salts), and C6.16.

4.6 Working Alone

Indicate which tasks of this project are of sufficiently low hazard that they may be carried out by a participant working alone, in particular off-hours. Alternately, it may be more convenient to list the tasks that are prohibited while working alone. Note if different rules apply to specific qualification levels among the participants (e.g., students).

The EMC's Working Alone Policy is included in the EMC101 EMC Users Orientation Course. The EMC has three levels of users. The status level of all active users is posted on the door to each instrument laboratory.

1. Microscope Trainees may not work alone: a supervising User must be present.
2. Microscope Users and Operators don't need to fulfill any special conditions to work alone in the electron microscope rooms during normal business hours (0730-1730, Monday through Friday).
3. Users who have demonstrated competence over time in operation of the instruments and who have proven that they can work safely are promoted by the EMC staff to Operator status. Only Operators are permitted to use the microscopes off-hours.

The use of computers to download/transfer data may be accomplished at any time by any level of user.

Forbidden activities:
1. Working with with liquid helium.
2. Working in the IVEM-Tandem facility if EMC staff members are not present.

Other work that the EMC evaluates to be outside the scope of this SAF will require a determination about whether the microscopist(s) will be allowed to work alone.

5. Feedback (ISM Core Function 5)

5.1 Records Kept

Identify types of records kept with this project that are useful in recreating and improving on the tasks within this project. In particular, include types of records that can be consulted if a task is unsuccessful or produces an unexpected result (in the scientific or operational sense). This could include lab notebooks, datasheets, computer data, instrument logs, images, etc.

Task/Situation	Record Kept
Nominal conditions of instrument	Instrument User Log: user name, specimen material, nominal operating conditions, specimen holders used, detectors used, etc.
Instrument use statistics	Electronic calendar
Experimental details	Individual investigator log books (non-EMC records)
Repairs/upgrades/maintenance	Instrument service log books (EMC use only)

5.2 Reporting

It is understood that technical results are reported to the outside world in scientific publications, presentations, and technical reports, and to the sponsor in program reviews, contractor meetings, and progress reports. Identify here the channels utilized to report the *operational* experience within the project, division, Argonne, or across the DoE complex. This should include emergency notifications, line management notifications, Lessons Learned (good or bad), group meetings (may serve as pre- or post-job briefings) and other communication channels.

Emergency	Call 911. Then notify the following people. 1. the lab custodian (first name on the room contact card) 2. the building manager, John Herman (2-6348; 630-918-9784; jherman@anl.gov) 3. the EMC director, Dean Miller (2-4108; 630-484-8841; 630-241-0674; miller@anl.gov) 4. your supervisor 5. the MSD division director, Michael Pellin (2-3510; 630-400-5309; 630-357-7572; pellin@anl.gov). 6. the MSD ESH coordinator, Urs Geiser (2-3509; 630-310-9443; 630-515-1712; ugeiser@anl.gov).
Unplanned events or unexpected results that could affect worker safety and health, the environment, the general public, or Argonne's reputation.	Stop work if danger is imminent. Then call the following people. 1. the lab custodian (first name on the room contact card). 2. the EMC director, Dean Miller (2-4108; 630-484-8841; 630-241-0674; miller@anl.gov) 3. If you cannot find either the lab custodian or Dean Miller, keep calling other EMC staff members until you find someone to help you.
Non-emergency but off-normal conditions	Contact EMC staff and record the problem in the instrument's User Log. EMC staff will investigate and document the problem and its solution in an instrument service log book.
User experience	Annual user survey

6. Radioactive Materials Summary

This section to be filled out only if this project utilizes radioactive materials

What isotopes and amounts will be involved?

Isotope	Physical Form[a]	Total Quantity Involved[b]	Quantity Typically Used in a Single Experiment[b]

[a] Physical form could be salt, powder, liquid, gas, etc.
[b] Specify units (dis/min, Ci, etc.).

Provide a schedule for the necessary radiation monitoring.

Where will the experiment be performed? (Identify all laboratories to be used, as well as hoods and/or glove boxes.)

What special provisions will be made for waste disposal?

Are additional or modified emergency plans required? ________ Yes ________ No

If so, identify appropriate changes and additions.

Will the experiment involve special nuclear materials? ________ Yes ________ No

Has the appropriate signage for experimental areas been approved by Health Physics? ________ Yes ________ No

Have radiation monitors been ordered for all researchers? ________ Yes ________ No

Estimate the total external radiation dose equivalents from this work (in person-rems): ________________________

Health Physics Name Signature Date

7. Certification, Review and Approval

7.1 Certification

It is my belief that I have identified all the hazards relating to this work, and that by following the procedures outlined above the Materials Science Division and Argonne National Laboratory will be exposed to an acceptable level of risk. I will make this document available to all participants of the project.

Dean J. Miller	4/16/2009
Signature, Principal Investigator	Date

7.2 Reviewers and Review Comments

List reviewers for this project and indicate (co-)coordinator/chair(s)

Stephan Rosenkranz, Chair	
Sam Bader, Co-chair	
Ursula Perez-Salas	

Hazard level and review process used: High hazard/complexity X Low hazard/complexity ____

Review team comment

7.3 Environmental Compliance (NEPA)

The NEPA review is usually carried out in conjunction with the funding proposal that supports this project, prior to this safety review. Environmental Compliance Representative (ECR) comment:

All environmental aspects are properly addressed. NEPA owner has been engaged for an ERF for the decommissioning of the VG603Z microscope.

Urs Geiser		4/16/2009
ECR Name	Signature	Date

7.4 Approvals and Authorization

The review team has reviewed the safety of this project and recommends its approval:

Stephan Rosenkranz	4/16/2009	Sam Bader	4/16/2009
Chair/Co-chair signature	Date	Co-chair signature	Date

Division director check one:

Approval of this project safety review authorizes this work to begin	X	
Separate work authorization is required (specify):		

I approve this project safety review:

Michael Pellin		4/16/2009
Division Director	Signature	Date